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REDSTONE ARSENAL RESEARCH DIVISION
HUNTSVILLE, ALABAMA

SPECIAL REPORT NO. S-79.

THE EXPLOSIVE CHARACTERISTICS OF
NF COMPOUNDS AND PROPELLANTS (U)

U.S. ARMY MISSILE COMMAND
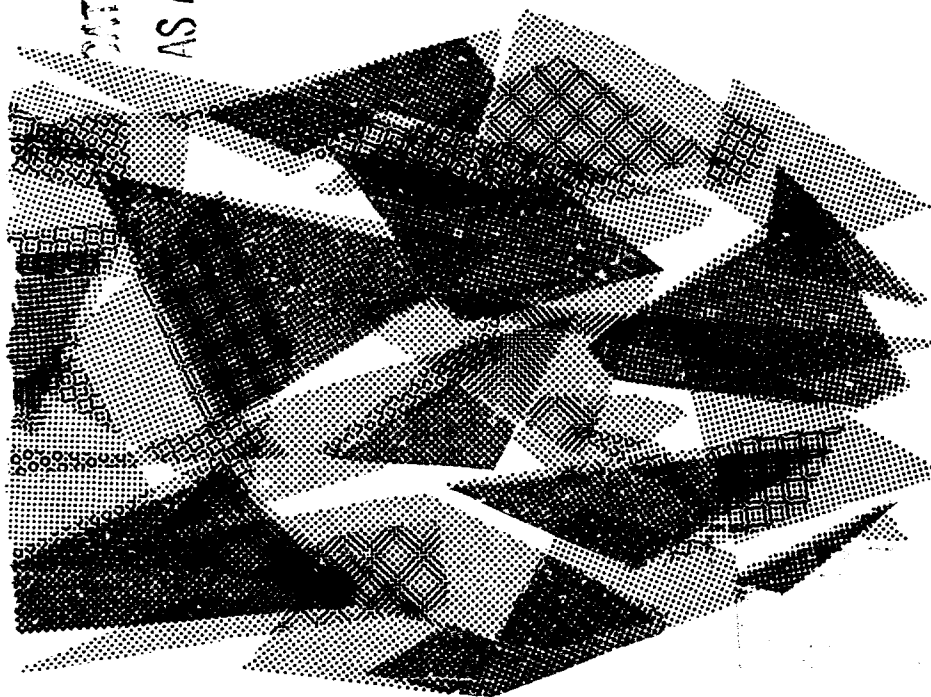
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HUNTSVILLE, ALABAMA

REPORT NO. S-79

THE EXPLOSIVE CHARACTERISTICS OF NF
COMPOUNDS AND PROPELLANTS

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THE EXPLOSIVE CHARACTERISTICS OF NF
COMPOUNDS AND PROPELLANTS

ABSTRACT

Difluoramino-substituted compounds are a class of high-energy substances which possess explosive properties. Composite propellants incorporating them in concentrations giving formulations of practical ballistic application lie within the current legal hazards category of Military Class 7.

This report contains all the card-gap-sensitivity and failure-diameter data obtained by these Laboratories, together with summaries of results of impact, differential-thermal-analysis, bottle-drop, ignition, friction, and electrostatic tests.

ACKNOWLEDGMENTS

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TABLE OF CONTENTS

|---|---|
INTRODUCTION	1
CARD-GAP-SENSITIVITY TESTS	3
The Small-Scale Card-Gap Test	3
Propellants	7
Liquids	12
FAILURE-DIAMETER DETERMINATIONS	16
DIFFERENTIAL THERMAL ANALYSIS	18
IMPACT-SENSITIVITY TESTS	20
IGNITION TESTS	22
BOTTLE-DROP TESTS	24
FRICTION- AND ELECTROSTATIC-SENSITIVITY TESTS	25
CONCLUSION	28
REFERENCES	29
APPENDIX A: SENSITIVITY DATA SUMMARY	
APPENDIX B: ABBREVIATIONS, ACRONYMS, AND CODES	

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INTRODUCTION

Throughout its research on the synthesis of energetic compounds
and development of high-performance propellants these Laboratories
have maintained continuous concern with and testing for the explosive
sensitivity of potentially hazardous materials, whether they be ingredients,
and intermediates (1, 2, 3, 4, 5),* or finished final propellant formulations
(5, 6, 7, 8). Although heavy reliance has been placed on card-gap tests
(8, 9, 10, 11), with which this report is primarily concerned, many
others are regularly employed. Among these are failure-diameter tests
(1, 7, 8), differential thermal analysis (12), impact tests (13), ignition
tests (3), bottle-drop tests (3), friction tests (14), and electrostatic
tests (14). In this report are assembled those sensitivity data which
have been accumulated on compounds and propellant formulations
containing difluoramino functional groups. Of the large amount of work
on related NF compounds performed by other organizations (11, 15),
the most pertinent results have also been included (16, 17).

The opportunity of examining NF formulations was created during
the ballistic evaluation of NF propellants. During this program it was
desirable to schedule an adequate amount of NF-containing material to
perform the tests of interest. Generally the results of the tests were
used for hazard evaluation. In the case of the card-gap tests, however,
an attempt at a systematic investigation was made to explore the possibility
of formulating a Military Class 2 (1?) NF propellant. Impact tests and
differential thermal analysis have been used as quality-control guides;
the latter has been helpful in investigating the thermal behavior

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*Numbers in parentheses indicate References listed at end of report.

of compounds of interest, ard, in some cases, in determining Arrhenius-
type apparent activation energies.

The description and discussion of results have been collected
under the indicated tests.

CARD-GAP-SENSITIVITY TESTS

The Small-Scale Card-Gap Test

The limited quantities of NF compounds available made it necessary to devise a small-scale card-gap test (10) so that a determination could be made on small amounts of sample. Since it was also desirable that such a small-scale test should yield a result from which could be inferred the value which would be obtained from the "standard" card-gap test (18), the test used was one in which only the acceptor and witness plate were reduced in size; the donor and barrier remained the same. The premise was that, with a given gap and explosive train, the card-gap value would not be strongly dependent upon the acceptor diameter.

The acceptor configuration was that of a sample contained in a 2.5-in. length of nominal $\frac{3}{8}$-in. Schedule 40 steel water pipe; the witness plate was a 3-in. $\times$ 3-in. sheet of $\frac{1}{8}$-in. mild steel (Fig. 1). A series of substances was then tested in each configuration* and a comparison was made (4); where possible, the samples were taken from the same batch in order to avoid batch-to-batch variation (Table I).

The two tests correlate well (Fig. 2). The small-scale test gives card-gap values 0.08 in. to 0.10 in. higher, on the average, than the large-scale test. Exceptions are seen in the case of RH-P-197 slurry at room temperature and Composition C-4 explosive. Nevertheless, the heavy preponderance of the remaining points support the validity, at least for cured propellants, of small-scale tests as good approximations to the large-scale card-gap results.

To pursue the differences further, a series of tests with familiar substances was performed in which the acceptors were placed off-center. The card-gap value decreased as the acceptors were displaced from the axis (Table II). These results indicated that there is curvature to the

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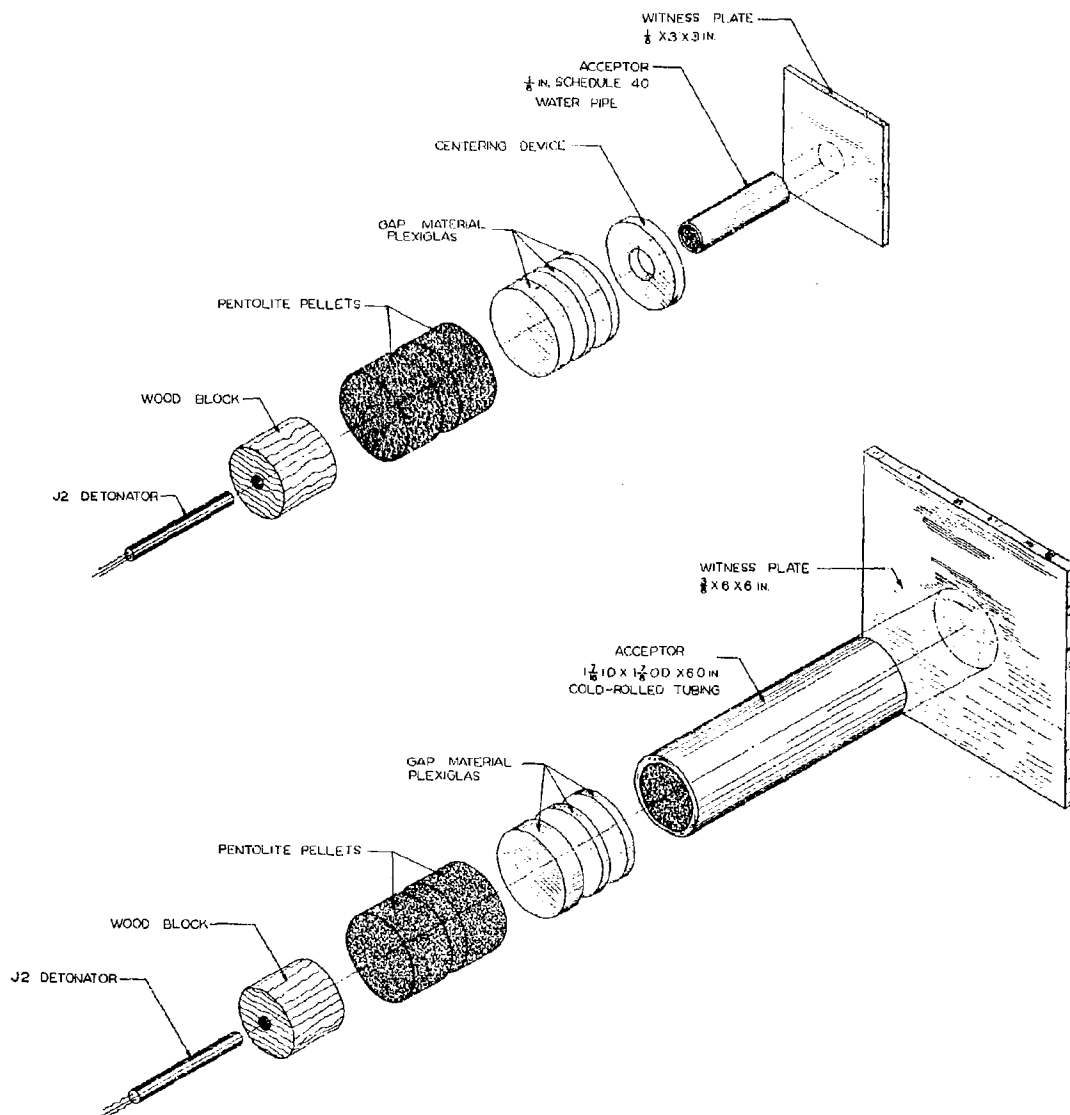*Referred to hereafter as small- and large-scale card-gap tests.

FIG. 1   CARD-GAP-TEST CONFIGURATIONS: SMALL-SCALE (TOP)
AND LARGE-SCALE (BOTTOM).

Table I

Comparison of Small-Scale and Large-Scale Card-Gap Values

Designation	Batch	Small-Scale Gap Thickness, in.	Large-Scale Gap Thickness, in.	Remarks
		Cured Propellants		
RH-P-112	1660	0.70 - 0.72	0.62 - 0.64	
RH-P-112	1772	0.81 - 0.86	0.66 - 0.68	
RH-P-112	1763	0.76 - 0.78	0.70 - 0.72	
RH-P-112	1772	0.78 - 0.80	0.72 - 0.74	
RH-P-181	a	0.46 - 0.48	0.42 - 0.44	
RH-P-197	1014	1.20 - 1.22	1.20 - 1.22	
RH-P-197	1016	1.20 - 1.22	1.24 - 1.26	
RH-P-324	1015	0.86 - 0.88	0.79 - 0.81	
RH-P-325	1001	1.06 - 1.08	0.90 - 0.92	
RH-P-387	1000	0.82 - 0.84	0.74 - 0.76	
RH-P-388	1000	0.64 - 0.66	0.56 - 0.58	
RH-SA-103	a	1.16 - 1.18	0.98 - 1.00	
LPC-1014	b	1.10 - 1.12	0.92 - 0.94	
		Slurries		
RH-P-112	1721	1.40 - 1.44	1.20 - 1.22	-85°F
RH-P-112	1721	1.50 - 1.56	1.27 - 1.32	-40°F
RH-P-112	1721	1.54 - 1.56	1.40 - 1.42	ambient
RH-P-197	1016	1.44 - 1.46	1.40 - 1.42	-76°F
RH-P-197	1016	1.46 - 1.51	1.42 - 1.47	-40°F
RH-P-197	1016	1.53 - 1.55	1.98 - 2.00	ambient
RH-SA-103	a	1.38 - 1.40	1.32 - 1.35	ambient
		Liquids		
TMETN	CL-6	0.29 - 0.30	0.28 - 0.29	77°F
NG/EG (3/1)	a	0.50 - 0.51	0.40 - 0.42	77°F

[a] acceptors from different batches.
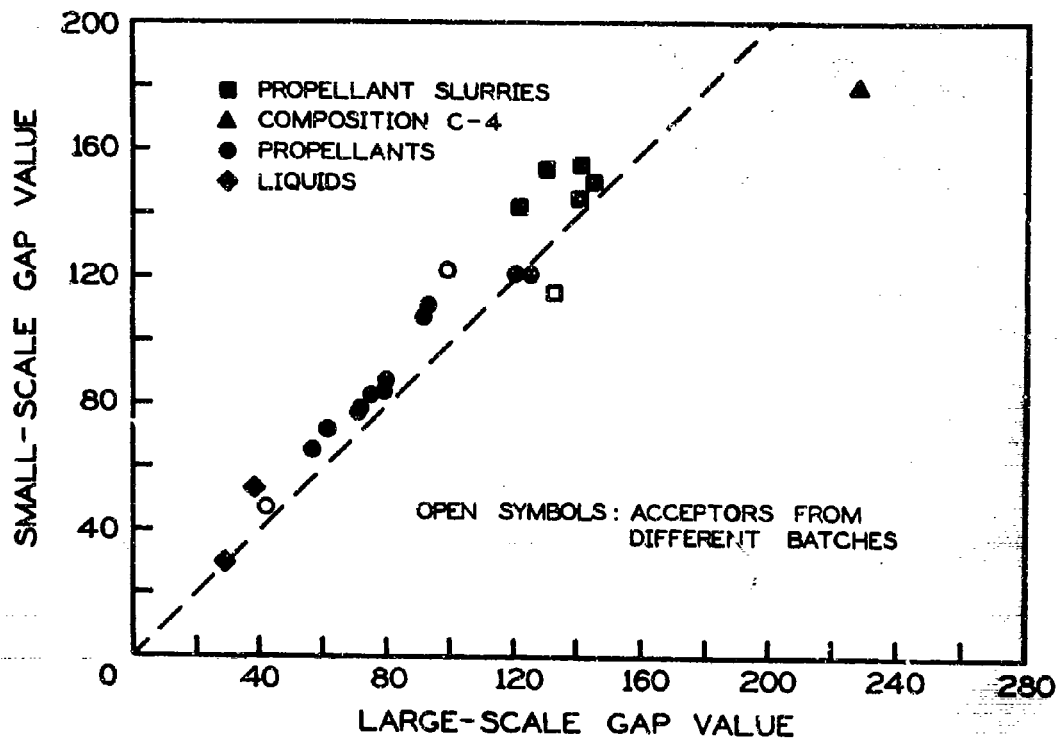[b] Rohm & Haas Company formulation, slightly modified, of a Lockheed Propulsion Co. propellant.

FIG. 2 COMPARISON OF SMALL-SCALE AND LARGE-SCALE
        CARD-GAP VALUES.

Table II

Change in Small-Scale Card-Gap Value with

Displacement of Acceptor

Axial Displacement, in.	Gap Thickness, in.
RH-P-112cb	
0	0.78 - 0.80
$\frac{7}{16}$	0.68 - 0.70
$\frac{11}{16}$[a]	0.46 - 0.48
Composition C-4 (ρ = 1.61 gm/cc)	
0	1.74 - 1.76
$\frac{7}{16}$	1.48 - 1.50
$\frac{11}{16}$[a]	1.34 - 1.36

[a]Maximum displacement on 2-in.-diam., donor subtending full acceptor diam.

shock front as it traverses the gap material; therefore, it is necessary to align the acceptor axially with the donor and gap material in order to obtain the best reproducibility.

Propellants

The small-scale card-gap test has been used to examine 27 NF propellants. For the most part these had been formulated for ballistic evaluation and were examined in the gap test in adjunct experiments. However, some significant trends of sensitivity effects can be sorted out.

At the outset it should be mentioned that, as a class, NF propellants exhibit high card-gap values compared with other types of propellants, and that the trends observed amount to changes which are small relative to the card-gap sensitivity level. For example, NF propellants have much higher card gap values than conventional plastisol-nitrocellulose composite propellants; they compare more closely with the RDX-containing plastisol-nitrocellulose propellants (Table III).

Table III

Comparison of Propellant Types in Small-Scale Card-Gap Tests

Designation	Type	Gap, in.
RH-SA-103	NF-monomer process	1.23
RH-SB-103	NF-prepolymer process	1.19
RH-P-112	Plastisol-nitrocellulose	0.71
RH-P-197	Plastisol-nitrocellulose + RDX	1.29

Several formulational variations have been examined. An increase in the plasticizer/monomer ratio in the TVOPA/NFPA* propellants increases somewhat the shock sensitivity of the resulting propellants (Table IV).

Table IV

Effect of Plasticizer/Monomer Ratios on Small-Scale
Values of NF Propellants

Designation	APC/Al/TVOPA/NFPA	Gap, in.
RH-SA-100	55/15/15/15	1.07
RH-SA-103	46/15/26/13	1.23
RH-SA-106	46/14/30/10	1.37

Shock sensitivity is not a function of ammonium perchlorate concentration at practical concentration levels (Table V), but it is a

Table V

Effect of Ammonium Perchlorate Concentration on Small-Scale
Card-Gap Values of NF Propellants

Designation	APC/Al/TVOPA/NFPA	Gap, in.
RH-SA-154	40/15/30/15	1.23
RH-SA-103	46/15/26/13	1.23
RH-SA-116	55/15/20/10	1.19

direct function of aluminum concentration (Table VI). A similar

*See Appendix B for definition of abbreviations.

Table VI

Effect of Aluminum Concentration on Small-Scale Card-Gap

Values of NF Propellants

Designation	APC/Al/TVOPA/NFPA	Gap, in.
RH-SA-168	61/0/26/13	0.93
RH-SA-167	54/7/26/13	1.15
RH-SA-103	46/15/26/13	1.23

dependence of sensitivity on aluminum concentration has been found in the plastisol-nitrocellulose propellants (19). In any case, a change in the ratio of constituents for APC-Al-TVOPA-NFPA propellants does not lower the shock sensitivity sufficiently to promise the successful formulation of a practical Military Class 2 propellant. Nor is the shock sensitivity appreciably reduced by diluting, in reasonable amounts, the plasticizer with an insensitive or inert substance (Table VII).

Table VII

Effect of Plasticizer Dilution on Small-Scale Card-Gap

Values of NF Propellants

Designation	APC/Al/TVOPA/DOS/NFPA	Gap, in.
RH-SA-140	49.0/16.0/16.6/0.9/17.5	1.13
RH-SA-142	49.7/15.3/14.9/2.6/17.5	1.03
RH-SA-141	49.7/15.3/19.8/3.6/11.6	1.09

Several compounds which held promise as alternate plasticizers or monomers were incorporated in propellants. However, for practical propellant compositions, there was no large shift in card-gap sensitivity for any of the compounds (Tables VIII and IX).

One NF propellant was formulated to accommodate a large-particle-size novel oxidizer. The card-gap sensitivity of this propellant was evaluated separately with ammonium perchlorate and nitronium perchlorate. The shock sensitivity of the two propellants was the same (Table X).

Table VIII

Effect of Plasticizer Variations on Small-Scale

Card-Gap Values of NF Propellants

Designation	APC/Al/Plasticizer/NFPA	Gap, in.
RH-SA-103	46/15/26 (TVOPA)/13	1.23
RH-SA-129	55/15/15 (HDN)/15	1.16
RH-SA-134	46/14/30 (HDN)/10	1.37
RH-SA-135	46/14/30 (TVOEA)/10	1.25
RH-SA-156	46/15/26 (HPE)/13	1.29

Table IX

Effect of Monomer Variations on Small-Scale

Card-Gap Values of NF Propellants

Designation	APC/Al/TVOPA/Monomer	Gap, in.
RH-SA-100	55/15/15/15 (vic-NFPA)	1.07
RH-SA-137	55/15/15/15 (gem-NFPA)	1.03
RH-SA-204	46/14/20/20 (TNFPA)	1.39
RH-SM-103	46/15/26/13 (NFPMA)	1.09
MAS-1	48/20/28/4 (MA)	1.45

Table X

Effect of Oxidizer Variations on Small-Scale

Card-Gap Values of NF Propellants

Designation	Oxidizer/Al/TNFH/K-120	Gap, in.
RH-SC-16	50/10/30/10 1000μ APC	0.95
RH-SC-17	50/10/30/10 700μ NP, 5% RETA-coated	0.91

The formulations discussed thus far had been made by a monomer process in which all the polymerization takes place during cure. Propellant so produced underwent a large degree of shrinkage during cure, undesirable

in casting grains in case-bonded motors. To reduce this shrinkage the
propellant was made by a prepolymer process in which NFPA was
copolymerized with a second monomer, usually HPMA, having chemically
active sites. The propellant-curing process then was reduced to cross-
linking the copolymer (or prepolymer) with a bifunctional compound, a
polyisocyanate in the case of HPMA. Propellant made by the prepolymer
process had card-gap sensitivities comparable to those made by the
monomer process (compare RH-SB-103 in Table XI with RH-SA-103 in

Table XI

Comparison of TVOPA and OPE in NFPA-Prepolymer Propellants

in Small-Scale Card-Gap Tests

Designation	APC/Al/Plasticizer/NFPA	Gap, in.
RH-SB-103	46/15/26 (TVOPA)/13	1.19
RH-SB-164	46/15/26 (OPE)/13	1.27
RH-SB-174[a]	40/10/37 (OPE)/13	1.59

[a]Maximum I_{sp}^* formulation

Table IV), and allows comparison of OPE with plasticizers itemized in
Table VIII.

Since it had been found that plastisol-nitrocellulose-propellant
slurries exhibited much higher card-gap values than their cured
counterparts (2), NF-propellant slurries were examined to determine
whether there was a similar difference; tests of NF slurries were also
of interest from the viewpoint of hazard evaluation. The NF slurries
had shock sensitivities comparable to those of their cured counterparts
(Table XII). It should be emphasized that the levels are the same as
those of plastisol-nitrocellulose propellant slurries.

Table XII

Small-Scale Card-Gap Values of Propellant Slurries

Designation	Type	Gap, in.
RH-SA-103	NFPA-monomer process	1.39
RH-SB-103	NFPA-prepolymer process	1.15
RH-P-112	Plastisol-nitrocellulose	1.55
RH-P-197	Plastisol-nitrocellulose + RDX	1.51

Liquids

The small-scale card-gap test has also been used to examine NF liquids, even though its significance as applied to liquids is in question (20). Such question is well raised, since it has been confirmed that the card-gap value for liquids in the small-scale test is a function of acceptor length (Table XIII). Moreover, it should be borne in mind that

Table XIII

Change in Small-Scale Card-Gap Sensitivity with Acceptor Length

Designation	Acceptor Length, in.	Gap, in.
NG/EG (3/1)	2.5	0.57-0.58
NG/EG (3/1)	16	0.32-0.34
TMETN	2.5	0.29-0.30
TMETN	16	0.18-0.20

the small-scale card-gap test referred to here is an adaptation of the standard test for solids (18) and is not a small-scale version of the standard test for liquids (21).

Several NF liquids have been examined along with familiar nitrate esters for comparison. NF liquids exhibit card-gap-sensitivity values higher than that of nitroglycerin (Table XIV). Among NF compounds studied, NFPA stands higher than TVOPA, contrary to

Table XIV

Small-Scale Card-Gap Values of Some Neat Liquids

Designation	Gap, in.
TMETN	0.30
NG/EG (3/1)	0.51
NG	0.91
TVOPA	1.05
OPE	1.03
HPE	1.19
NFPA (monomer)	1.21

results of most other kinds of sensitivity and stability tests; the only other instance in which NFPA looks more sensitive than TVOPA is by the differential-thermal-analysis test (q. v.) (12).

An intermediate in the manufacture of an NF propellant is a mixture of the plasticizer and the prepolymer (or monomer). Accordingly, these mixtures were subjected to the card-gap test. A 2/1 mixture of TVOPA/NFPA monomer gave a card gap value of 1.17 in.; a 2/1 mixture of TVOPA/NFPA prepolymer had a card gap value of only 0.49 in. This was the first large reduction in card-gap sensitivity of NF compounds ever to be observed.

To sort out the reason(s) for such a reduction, two additional determinations were made (Table XV). The first was made on a TVOPA

Table XV

Small-Scale Card-Gap Values of Some Liquid Mixtures (2/1)

Containing TVOPA

Designation	Gap, in.
TVOPA/NFPA [monomer]	1.17
TVOPA/(NFPA/HPMA[9/1]) [prepolymer]	0.49
TVOPA/(NFPA/HPMA[9/1]) [ternary mixture]	0.99
TVOPA/APA	0.53

solution of unprepolymerized NFPA and HPMA with the constituents in the same ratio as the prepolymer. The card-gap value for this ternary solution was 0.99 in.; the 0.18-in. reduction in gap thickness could easily have been due to simple dilution by the HPMA.

The second determination was made on a 2/1 solution of TVOPA in the saturated analog of NFPA, APA, to investigate the significance of the double bond. The card-gap value of the latter mixture was 0.53 in., indicating that the presence of the double bond did indeed hold the key to sensitization of the solution. This hypothesis was not vindicated, however, when NFPA prepolymer was tested with OPE and HPE plasticizers in similar solutions. The NFPA prepolymer did not desensitize OPE to the same degree as it apparently desensitized the TVOPA, nor HPE at all (compare Tables XIV and XVI).

Table XVI

Small-Scale Card-Gap Values of Some Liquid Mixtures (2/1)

with NFPA Prepolymer

Designation	Gap, in.
TVOPA/NFPA	0.49
OPE/NFPA	0.75
HPE/NFPA	1.11

The possibility that the card-gap value for the TVOPA–NFPA-prepolymer solution was an artifact of the experiment (viz. the failure diameter was so close to the diameter of the acceptor as used in the small-scale card-gap test as to give a spurious low value, as has been found with solid explosives) was investigated. The card-gap value, with the acceptors confined in nominal $3/4$-in. Schedule 40 water pipe, 0.82 in. I. D., with the usual explosive–gap-material train, was 0.50 to 0.60 in., showing that the value of 0.49 in. in the small-scale test was probably real.

In summary, the small-scale card-gap test has shown that NF propellants of the types which have been examined thus far will be Military Class 7. It has also provided a body of data on NF compounds which indicate that the intermediates used in the manufacture of NF propellants are sensitive substances from the standpoint of explosive-shock initiation. The complete body of data are given in Appendix A.

FAILURE-DIAMETER DETERMINATIONS

The failure diameter, also termed critical diameter or minimum diameter, is defined as that diameter of a cylindrical charge below which steady high-velocity detonation cannot propagate in the medium (8). It is a function of both the chemical and physical nature of the test substance as well as the degree of charge confinement. It is usually determined experimentally between limits by firing a number of rounds of various diameters with unattenuated high-explosive donors having diameters at least as great as those of the test rounds. The condition of witness plates at the downstream end of the charge is the most common criterion of whether a detonation has occurred, although velocity measurements are often employed.

A limited number of failure-diameter determinations on NF propellants and compounds have been made. Those performed on propellants were made in the beginning phase of the program developing the small-scale card-gap test, to insure that the 0.48-in. acceptor size chosen would lie above the failure diameters of the substances to be ranked in this test. Failure diameters larger than 0.48 in. were rare among the propellants of interest. One such exception was RH-SA-1, which exhibited a failure diameter of greater than 0.75 in. in steel confinement (Table XVII).

Failure-diameter determinations for liquid mixtures of TVOPA and DCE have also been made. A 1/1 mixture of TVOPA to DCE gave a value of $0.48 < D_f < 0.62$, while the 2/1 mixture gave $D_f < 0.48$. This result merely shows the magnitude of the increase in failure diameter with the addition of diluent.

It should be pointed out that any substance for which a small-scale card-gap value can be obtained must ipso facto have a failure diameter of less than 0.48 in. in steel confinement. Therefore this upper limit applies to HPE, OPE, etc., whose small-scale card-gap data are given in the previous section.

Table XVII

Failure Diameters of NF Propellants and Ingredients

(Steel Confinement[a])

Designation	Batch	D_f, in.	Remarks
		Propellants	
RH-SA-1	01	>0.75	APC/Al/NFPA = 53/12/35
RH-SA-100	1003	0.27 - 0.36	APC/Al/TVOPA/NFPA = 55/15/15/15
RH-SA-103	1013	0.36 - 0.48	APC/Al/TVOPA/NFPA = 46/15/26/13
RH-SA-103	1055	<0.62	cardboard-confined
RH-SA-103	1060	<0.27	
RH-SA-106	1002	0.27 - 0.36	APC/Al/TVOPA/NFPA = 46/14/30/10
RH-SA-116	02	<0.27	APC/Al/TVOPA/NFPA = 55/15/20/10
RH-SA-142	01	0.25 - 0.36	APC/Al/TVOPA/DOS/NFPA = 50/15/15/3/17
RH-SM-103	1000	<0.27	APC/Al/TVOPA/NFPMA = 46/15/26/13
		Liquids	
TVOPA	573C	<0.48	
NFPA	564	<0.48	
TVOPA/DCE		<0.48	2/1
TVOPA/DCE		0.48 - 0.62	1/1

[a]Schedule 40 water pipe, L/D > 4

DIFFERENTIAL THERMAL ANALYSIS

Differential thermal analysis (12) has been used as a measure
of explosive sensitivity (3) on the principle that compounds with low
thermal stability in the environment of this test may well be easily
initiated to detonation. The merit of DTA tests lies in the small sample
required, which makes the tests useful in examining novel compounds
at the very beginning of its laboratory development. Along with
impact tests, DTA can serve early warning of potential hazards to be
met.

It is noteworthy that, of all the kinds of tests described in the
present Report, only DTA (Table XVIII) presaged the relative ranking
of NFPA above TVOPA in the card-gap test.

For a full discussion of data obtained at these Laboratories,
see Ref. 12.

Table XVIII

Summary of Differential-Thermal-Analysis Data

(K. G. Scrogham)

Designation	Peak Exotherm,[a] °C
Liquids	
TVOPA	265
APA	185[b]
OPE	239
HDN	211
TNFH	205[b]
NG	196
Polymers	
NFPA (monomer)	225[c]
TAMA (monomer)	216[c]
NFPMA (polymer)	230
NFPA/HPMA = 90/10 (copolymer)	190
NFPMA/HPMA = 90/10 (copolymer)	214
NFPA/IA = 90/10 (copolymer)	231
Mixtures	
TVOPA/APA = 2/1	264
TVOPA/NFPA (monomer) = 2/1	234
Propellants[d]	
RH-P-112	173
RH-SA-100	252
RH-SA-103	259
RH-SB-103	244
RH-SA-151[e]	246
RH-SB-164	216

[a] Heating rate = 10C/min.
[b] Endotherm at boiling point.
[c] Preceded by a polymerization peak, see Ref. 12.
[d] See tables of card-gap values for formulations.
[e] APC/Al/HPE/NFPA = 55/15/15/15

IMPACT-SENSITIVITY TESTS

Impact-sensitivity testing (13) has been done at these Laboratories on a routine basis for a number of years employing both the modified Picatinny machine and occasionally the so-called Olin-Mathieson or liquid machine. Since the test can be performed on a small quantity of material, it is one of the first to be applied to a novel compound, along with differential thermal analysis. As with DTA, the results of the tests are used as a qualitative foretoken of the potential hazard of new materials. Impact tests are also performed on intermediates and cured propellants as criteria for hazard evaluation.

A representative sample of impact sensitivity is given in Table XIX. These results were obtained with the Picatinny machine, either on finely divided solids or on slurries and liquids on filter paper.

Table XIX

Selected Impact-Sensitivity Data

(F. A. Johnson)

Sample	Drop Height[a], in., No. of fires/No. of trials										Calculated 50% Point, in.
	45.9	32.4	22.8	16.1	11.3	8.0	5.6	4.0	2.8	1.9	
					Group I[b]						
TMETN	2/5	5/8	5/11	4/11	0/5						22.2
NC/EG (60/40)			2/2	8/10	10/18	1/10					11.7
NG[c]			4/4	5/10	5/11	3/8	2/5	0/2			10.7
TEGDN	0/10	0/1	0/1	0/1	0/1						>38
TVOPA (500)		3/3	10/13	4/14	1/5	1/2	1/2	0/1			15.5
TVOPA (573B)			2/2	4/7	12/7	1/13	0/1				10.9
NFPA	0/10	0/1	0/1	0/1	0/1						>38
					Group II[b,d]						
RH-SB-193 slurry						4/4	1/5	0/1			6.3
RH-SB-174 slurry						1/1	1/1	2/2	3/4	0/2	3.0
RH-SB-174							3/3	2/5	2/5	0/2	3.4
RH-P-112							5/5	0/5			6.7
OPE					1/1	1/1	1/1	1/1	3/3	0/3	2.5
TP-3 (1008)	3/3	1/3	0/1	0/1	0/1	0/1					36.8
					Group III[e]						
RH-SA-100						1/1	4/5	0/4			7.2
RH-SA-106							3/3	2/5	0/2		8.3
RH-SA-129						1/1	2/3	2/4	0/2		6.3
RH-SA-133[f]							5/5	0/5			9.5
RH-SA-134							5/5	0/5			6.7
RH-SA-135						1/1	4/5	0/4			7.2
RH-SA-203[g]							3/3	2/5	0/2		5.8

[a] Samples randomized and increments selected by Bruceton up-and-down method
[b] Picatinny impact, kg-in., 1-kg. weight; 50% level for RDX =12.9 in.
[c] Stripped at 0.2 mm. Hg for 4 hours.
[d] Samples in this group were tested in conjunction with friction tests performed by Thiokol Chemical Corporation. Tests were done in the same manner as Group I, but were performed at a different time.
[e] 2-Kg weight; 50% level for RDX = 9 in.
[f] APC/Al/TVOEA/NFPA = 55/15/15/15
[g] APC/Al/TVOPA/TNFPA = 55/15/15/15

IGNITION TESTS

The properties of materials in bulk subjected to thermal initiation cannot be inferred from tests described up to this point. The shock tests impose more severe stimulus than is likely to be encountered in actual processing and handling; impact-sensitivity and DTA tests utilize only a few drops of sample per shot.

A rudimentary test has been improvised in which liquids are exposed to thermal ignition in both the liquid itself and the vapor region above the surface. The liquid sample is placed in a 600-ml. beaker to a depth of about $\frac{1}{4}$ in. (~25cc) and then exposed to simple flame (Atlas match) or thermal and percussive stimulus (squib). The following sequence of tests is made until reaction occurs:

    (1) an Atlas match at the surface of the liquid,

    (2) an Atlas match submerged in the liquid,

    (3) an M-1 squib at the surface of the liquid,

    (4) an M-1 squib submerged in the liquid.

Thus far, three degrees of reaction have been observed: explosions (with TVOPA), simple burning, and no ignition or observable reaction (Table XX).

The propensity of a liquid to transit from deflagration to detonation is a function, among other things, of the total bulk present and cannot be inferred from small-scale tests. Therefore, the results here serve to point out hazards only to a limited degree; failure of a substance to explode in this test gives no assurance that it is harmless in larger quantities.

Table XX

Ignition-Test Data

Designation	Batch	Test Results [a]			
		1	2	3	4
TVOPA		$Ex^{2/2}$			
TVOPA/DCE (2/1)		$NR^{0/1}$	$NR^{0/1}$	$NR^{0/1}$	$NR^{0/1}$
TVOPA/DCE (1/1)		$NR^{0/1}$	$NR^{0/1}$	$NR^{0/1}$	$NR^{0/1}$
NFPA		$NR^{0/1}$	$NR^{0/1}$	$NR^{0/1}$	$NR^{0/1}$
TVOPA/NFPA (2/1)		$NR^{0/1}$	$NR^{0/1}$	$B_s^{1/1}$	
TVOPA/NFPA (1/1)		$NR^{0/1}$	$NR^{0/1}$	$NR^{0/1}$	$NR^{0/1}$
TP-1	1000	$B_s^{2/2}$			
TP-8	1000	$B_s^{1/1}$			
NG/EG (2/1)		$NR^{3/3}$			
RH-SB-103cb slurry	MB-657-54	$B_p^{1/1}$			
RH-SB-103cd slurry	1075	$B_p^{1/1}$			$B_p^{1/1}$

[a] Test 1: Atlas match at surface
Test 2: Atlas match submerged
Test 3: M-1 squib at surface
Test 4: M-1 squib submerged
Ex: exploded
B_s: burned, leaving sooty residue
B_p: burned like propellant, leaving white residue
NR: no exothermic reaction observed

Superscripts give ratio of the no. of positive results to the total no. of trials.

BOTTLE-DROP TESTS

Since a quantity of material is much more likely to be dropped or have something dropped on it than it is to be subjected to an explosive shock, a bottle-drop test has also been devised at these Laboratories for hazard evaluation (3). The test merely consists of dropping a bottle onto a steel plate from various heights and observing the reaction, if any, which takes place.

Two test configurations have been employed. The first consists of dropping a 26-cc glass vial containing about 20 cc of test material; in this test, the bottle always breaks and on only one occasion has an explosion resulted — TVOPA from 10 feet. The second consists of placing a pint glass bottle, partially filled with the test sample, in a container lined with foam rubber. The container is dropped normal to a steel plate from varying heights, increased at intervals until either an event occurs or the test is terminated at 10 feet. The data from these tests are given in Table XXI.

Table XXI

Bottle-Drop-Test Data

(Dwyer, Minton, and Parrott)

Designation	Drop Height, feet								
	10	9	7	6	5	4	3	2	1
TVOPA[a]	Ex$^{1/3}$				NR$^{9/1}$				
TVOPA/DCE (2/1)[a]	NR$^{9/5}$								
TVOPA/NFPA (2/1)[a]	NR$^{9/1}$								
NG[a]	NR$^{9/5}$				NR$^{9/1}$				
TVOPA[b]		NR$^{9/4}$	NR$^{9/10}$	NR$^{9/10}$	NR$^{9/10}$	NR$^{9/5}$	NR$^{9/5}$	NR$^{9/5}$	NR$^{9/5}$

[a] Stoppered 26-cc glass vial with 20 cc of test material on to a steel plate.
[b] Stoppered 1-pint glass bottle, with 0.2 pint of liquid, contained in rubber-lined carrier; successive drops.
[c] Ex: Exploded
NR: No exothermic reaction observed.

Superscripts give ratio of the no. of positive results to the total no. of trials.

FRICTION- AND ELECTROSTATIC-SENSITIVITY TESTS

Since these Laboratories have no capability to perform friction-
or electrostatic-sensitivity tests on propellants or propellant ingredients,
Thiokol Chemical Corporation, Huntsville Division, has conducted a
limited number of tests on samples of propellant for us.

The friction test (14) consists of placing a thin sample, usually
0.020 in. thick, between a rotating disc and a fixed plate at a constant
pressure. The speed of the disc, in rpm, and the time to reaction
are taken as measures of the friction sensitivity. The limit of the
disc speed is 7,000 rpm and a test is termed a "no go" if no reaction
is observed in 120 seconds. The results show that, in this test, the
NF propellants tested are more friction-sensitive than the plastisol-
nitrocellulose propellants (Table XXII).

In conjunction with these tests, Thiokol Chemical Corporation
also performed electrostatic-sensitivity tests. It was found that
none of the propellants submitted for testing reacted at the test limit
of 20 joules. Neat TVOPA, NFPA, and a mixture of TVOPA/NFPA =
2/1 were also tested; the results were mixed (Table XXIII).

Table XXII

Friction-Sensitivity-Test Data

(Thiokol Chemical Corporation)

Designation	Disc Speed, rpm	Time, sec	Result
RH-SA-103	1500	40	+
	1400	60	+
	1200	120	-
	1200	20	+
	1200	50	+
	1100	30	+
	1100	50	+
	1000	120	-
	1000	120	-
RH-SB-103	1500	15	+
	1300	25	+
	1200	120	-
	1200	30	+
	1200	60	+
	1100	120	-
	1100	120	-
RH-SB-164	800	40	+
	600	70	+
	600	90	+
	500	120	-
	500	120	-
RH-SA-156	1000	12	+
	600	45	+
	500	25	+
	500	50	+
	400	120	-
	400	120	-
RH-SB-174	800	25	+
	500	50	+
	500	65	+
	400	120	-
	400	120	-
RH-P-112	7000	120	-[a]
RH-P-197	7000	120	-[a]

[a] Limit of test.

Table XXIII

Electrostatic-Sensitivity-Test Data

(Thiokol Chemical Corporation)

Designation	Stimulus, joules	Result
RH-SA-103	20[a]	-
RH-SB-103	20	-
RH-SB-164	20	-
RH-SA-156	20	-
RH-P-197	20	-
TVOPA	1	-
TVOPA	2	+
NFPA	1	-
NFPA	2	+
TVOPA/NFPA (3/1)	10	-

[a]Limit of test.

CONCLUSION

As far as explosives classification is concerned, it is evident from card-gap tests alone that there is little promise of developing a Military Class 2 (fire hazard only) propellant based on difluoramino compounds, which propellant would also offer ballistic advantages over present state-of-the-art formulations.

As for hazard evaluation, all the data together cannot give a quantitative answer. Hazard evaluation is the result of a subjective appraisal. Such an appraisal has been made in the case of TVOPA, based on assembled data reproduced in Appendix Tables A-2, A-3, and A-4 (3); the conclusions reached are quoted below:

"1. TVOPA is a sensitive material which can react to release energy at a hazardous rate. TVOPA is apparently as sensitive as nitroglycerin to shock and impact initiation. In view of this TVOPA should continue to be handled carefully.

"2. TVOPA is thermally stable at temperatures greater than 100°C and, therefore, spontaneous reaction of TVOPA not subjected to external sources of initiation is improbable.

"3. TVOPA can be desensitized by dilution with a suitable solvent such as 1,2-dichloroethane."

Hazard appraisal is a continuing activity and incorporates not only the results of direct tests but also those of experience. Moreover, new methods of handling hazardous materials are continually being explored; among these are desensitization, dilution, and alteration of the chemical nature of the intermediates, such as further investigation of the prepolymer technique discussed in the preceding sections.

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Appendix A

SENSITIVITY DATA SUMMARY

Table A-I

Consolidated Small-Scale Card-Gap Data

(J-2-initiated, 160-gm, 2-in.-diam. X 2-in. pentolite donor; 2-in.-diam.
Plexiglas gap; 0.48-in.-I.D. X 2.5-in. Sch. 40 steel water-pipe acceptor confinement)

Designation	Batch	Gap,[a] in.	Remarks
		Propellants	
RH-SA-100	1000	0.98¹ - 1.22⁰	APC/Al/TVOPA/NFPA = 55/15/15/15
RH-SA-100	1003	1.06¹ - 1.08²	
RH-SA-103	05	1.16¹ - 1.18¹	APC/Al/TVOPA/NFPA = 46/15/26/13
RH-SA-103	1013	1.22² - 1.24⁴	Samples not completely cured.
RH-SA-103	1015	1.22² - 1.24⁰	
RH-SA-106	1001	1.36¹ - 1.38⁰	APC/Al/TVOPA/NFPA = 46/14/30/10
RH-SA-116	02	1.18² - 1.20⁰	APC/Al/TVOPA/NFPA = 55/15/20/10
RH-SA-129	02	1.15² - 1.17⁰	APC/Al/HDN/NFPA = 55/15/15/15
RH-SA-134	01	1.35² - 1.40⁰	APC/Al/HDN/NFPA = 46/14/30/10
RH-SA-135	01	1.24¹ - 1.26⁰	APC/Al/TVOEA/NFPA = 46/14/30/10
RH-SA-137	01	1.02¹ - 1.04⁰	APC/Al/TVOPA/gem-NFPA = 55/15/15/15
RH-SA-140	01	1.12¹ - 1.14⁰	APC/Al/TVOPA/DOS/NFPA = 49/16/17/1/17
RH-SA-141	01	1.08² - 1.10⁰	APC/Al/TVOPA/DOS/NFPA = 50/15/20/4/11
RH-SA-142	01	1.02² - 1.04⁰	APC/Al/TVOPA/DOS/NFPA = 50/15/15/3/17
RH-SA-145ci	06	0.95¹ - 1.22⁰	APC(5µ)/Al/TVOPA/NFPA = 40/10/38/12
RH-SA-147	01	0¹ - 1.00⁰	APC/Al/DGDEA/NFPA = 55/15/15/15
RH-SA-147	02	1.06¹ - 1.08⁰	
RH-SA-148	01	no go	APC/Al/AAA/NFPA = 55/15/15/15
RH-SA-154	01	1.22¹ - 1.24¹	APC/Al/TVOPA/NFPA = 40/15/30/15
RH-SA-156	04	1.27² - 1.30⁰	APC/Al/HPE/NFPA = 46/15/26/13
RH-SA-167	01	1.14² - 1.16⁰	APC/Al/TVOPA/NFPA = 54/7/26/13
RH-SA-168	01	0.92¹ - 0.94⁰	APC/Al/TVOPA/NFPA = 61/0/26/13
RH-SA-204	02	1.38⁶ - 1.40¹	APC/Al/TVOPA/TNFPA = 46/14/20/20
RH-SB-103	MB665-6	1.18¹ - 1.20⁰	APC/Al/TVOPA/PPNF-2 = 46/15/26/13
RH-SB-103cd	1062	1.15² - 1.18⁰	47µ APC
RH-SB-164	MB665-18	1.25¹ - 1.28¹	APC/Al/OPE/PPNF-2 = 46/15/26/13
RH-SB-174	MB1063-5	1.58¹ - 1.60⁰	APC/Al/OPE/PPNF-2 = 40/10/37/13
RH-SM-103	1000	1.08² - 1.10⁰	APC/Al/TVOPA/NFPMA = 46/15/26/13
RH-SC-16	01	0¹ - 0.48⁰	APC(16-20 mesh)/Al/TNFH/K-120 = 50/10/30/10
RH-SC-16	02	0.90¹ - 1.00⁰	
RH-SC-17	01	0.90¹ - 0.92⁰	NP(20-30 mesh, 5% RETA-coated)/Al/TNFH/K-120 = 50/10/30/10
MAS-1	01	1.44² - 1.46¹	APC(170µ)/APC(17µ)/Al/TVOPA/MA = 36/12/20/28/4
Binder		no go	TNFH/K-120 = 3/1

Table A-I continued

Designation	Batch	Gap,[a] in.	Remarks
			Slurries
RH-SA-103	31	$1.38^{2/6} - 1.40^{1/2}$	APC/Al/TVOPA/NFPA = 46/15/26/13
RH-SA-106	1002	$1.46^{2/6} - 1.48^{0/2}$	APC/Al/TVOPA/NFPA = 46/14/30/10
RH-SB-103	MB657-54	$1.14^{1/1} - 1.18^{0/2}$	APC/Al/TVOPA/PPNF-2 = 46/15/26/13
RH-P-112	1722	$1.54^{1/0} - 1.56^{0/1}$	APC/Al/TEGDN/DBP = 30/15/37/17
RH-P-197	1015	$1.50^{2/6} - 1.52^{0/1}$	APC/RDX/Al/TMETN/TEGDN/DBP = 10/29/18/24/8/10
			Liquids
IBA	615-2	$0.78^{1/6} - 0.80^{0/2}$	87.2% assay; 77°F
NFPA		$1.28^{2/6} - 1.31^{0/2}$	77°F
TVOPA		$1.20^{2/6} - 1.22^{0/2}$	77°F
TVOPA/NFPA		$1.00^{1/6} - 1.05^{0/1}$	2/1; 77°F
NFPA	730	$1.20^{0/1} - 1.22^{0/2}$	77°F
TVOPA	694	$1.04^{2/6} - 1.06^{0/2}$	83°F
TVOPA/NFPA	694/730	$1.16^{2/6} - 1.18^{0/2}$	2/1; 85°F
TP-1	1000	$0.46^{2/0} - 0.48^{0/2}$	77°F
TP-1	1001	$0.48^{2/6} - 0.50^{0/2}$	83°F
TP-8	1000	$0.54^{2/6} - 0.56^{0/2}$	87°F
TVOPA/NFPA/HPMA		$0.98^{1/6} - 1.00^{0/1}$	20/9/1
TVOPA/APA		$0.52^{1/0} - 0.55^{1/1}$	2/1
HPE		$1.15^{1/6} - 1.22^{0/1}$	Incomplete
HPE/PPNF-3		$1.10^{2/6} - 1.12^{0/5}$	2/1
OPE		$1.02^{1/6} - 1.05^{0/2}$	
OPE/PPNF-3		$0.74^{1/6} - 0.76^{0/1}$	2/1
NFPOH/CCl$_4$	733	no go	7/3; 80°F
NG		$0.90^{2/0} - 0.92^{1/2}$	77°F
NG/EG		$0.50^{3/6} - 0.51^{0/2}$	3/1; 77°F
TMETN	GL-6	$0.29^{3/1} - 0.30^{0/2}$	77°F

[a] Superscripts give ratio of go/no-go at indicated gap thickness. These sum to less than the total no. of trials; they indicate only the bounds of the 50% point.

Data complete through internal Entry No. 479
6/29/65

Table A-II

Summary of Hazard Assessment Data on TVOPA, NFPA, and NG

(Ref. 3)

	TVOPA	NFPA	NG
50% Picatinny impact, kg-in.	10.9	>38	10.7
Card gap, in.[a]	1.21	1.30	0.91
Ignition tests[b,c]			
Atlas match at surface	Ex[2/1]	NR[0/1]	--
Atlas match submerged	--	NR[0/1]	--
M-1 squib at surface	--	NR[0/1]	--
M-1 squib submerged	--	NR[0/1]	--
Bottle-drop test - 10 ft.[c,d]	Ex[1/3]	--	NR[0/3]
Differential thermal analysis[e]			
Start of exotherm, °C	160	115	146
Peak exotherm, °C	260	225	196

[a] These results were obtained in the small-scale card-gap test.
[b] Tests were run in open 600-cc. Pyrex beakers containing approximately 40 grams ($\frac{1}{4}$-inch depth) of the test material.
[c] Ex: Reacted with explosive violence
NR: No exothermic reaction observed
[d] This test consisted of dropping a glass vial (volume = 26 cc) containing 20 cc of the test material from the specified height and allowing it to impact on a steel plate.
[e] Heating rate = 10C°/min.

Table A-III

Sensitivity Data on TVOPA–1,2-Dichloroethane Solutions

	TVOPA	TVOPA/DCE (2/1)	TVOPA/DCE (1/1)
Minimum diameter (steel confinement) - in.	<0.484	<0.484	$0.484 < D_f < 0.622$
Bottle drop - frequency of explosive reaction	Ex$^{1/6}$	NR$^{0/6}$	--
50% Picatinny impact - kg-in.	9	15	30
Ignition tests			
Atlas match - surface	Ex$^{2/6}$	NR$^{0/4}$	NR$^{0/4}$
Atlas match - submerged	--	NR$^{0/4}$	NR$^{0/4}$
Squib - surface	--	NR$^{0/4}$	NR$^{0/4}$
Squib - submerged	--	NR$^{0/4}$	NR$^{0/4}$

Table A-IV

Du Pont Sensitivity Tests on TVOPA

(Ref. 16)

Test	Result[a]
Hot bar test	TVOPA evaporates on contact with bar at 250°C
Copper block heat test (Heating rate = 5C°/min)	No visible reaction up to maximum temperature of 250°C
Modified du Pont drop test[b]	50% point = 129 kg-cm.
8.3 g. ball drop test[c]	30-35 inches.
Base load test	#0[d]
Differential thermal analysis[e]	
Start of exotherm	11°C
Peak of exotherm	274°C
Static sensitivity	No Go at 30 kV & 10^4 picofarads or 4.5 joules[f]

[a] Test descriptions are given in detail in du Pont Report, Elab-D-171, pp. 42-47.

[b] NG: 5 kg-cm.; PETN: 318 kg-cm.

[c] Lead azide: 12 inches.

[d] This is the most positive reaction in this test which involves measuring the size of the hole in a witness plate resulting from the initiation of a standard quantity of the test explosive by a standard du Pont cap.

[e] Heating rate = 7.5C°/min.

[f] This is about 300 times the amount of electrical energy that can accumulate on an insulated man.

Appendix B

ABBREVIATIONS, ACRONYMS, AND CODES
(X = NF$_2$)

AAA   2, 3-Bis(difluoramino)propyl acetate
$CH_3COOCH_2CHXCH_2X$

Al   Aluminum
Alcoa 140, exceptions noted

APA   2, 3-Bis(difluoramino)propyl propionate
$C_2H_5COOCHCHXCH_2X$

APC   Ammonium perchlorate
cb grind, exceptions noted

BPO   Benzoyl peroxide
$(C_6H_4CO)_2O_2$

C-4   Composition C-4 explosive
91% RDX in synthetic wax

DBP   Double-base powder
"Fluid Ball"® Propellant [Olin Mathieson]

DCE   1, 2-Dichloroethane
CH_2ClCH_2Cl

DGDEA   1, 2, 10, 11-Tetrakis(difluoramino)-3, 6, 9-trioxaundecane
$(CH_2XCHXOCH_2CH_2 \cdot)_2O$

DOS   Dioctyl sebacate
$(CH_2)_8(COOC_8H_{17})_2$

EG   Ethylene glycol
$HOCH_2CH_2OH$

HPE   1, 2, 3, 5, 6, 7-Hexakis(difluoramino)-4-oxaheptane
$(CH_2XCHX \cdot)_2O$ [Esso]

HPMA   3-Hydroxypropyl methacrylate
$CH_2:C(CH_3)COOCH_2CH_2CH_2OH$

HDN   1, 2, 4, 5, 8, 9-Hexakis(difluoramino)nonane
$CH_2XCHXCH_2CHXCHXCH_2CH_2CHXCH_2X$

IA   Itaconic anhydride
$CH_2:C(CO)CH_2CO$ (with ─O─ ring)

IBA   1, 2-Bis(difluoramino)-2-methylpropane
$CH_2XCX(CH_3)_2$

K-120   Acrylic resin
Acryloid K-120® [Rohm & Haas]

MA   Methyl acrylate
            $CH_2:CHCOOCH_3$

NG   Nitroglycerin
            $O_2NOCH_2CH(ONO_2)CH_2ONO_2$

NFPA   2,3-Bis(difluoramino)propyl acrylate
            $CH_2:CHCOOCH_2CHXCH_2X$

gem-NFPA   2,2-Bis(difluoramino)propyl acrylate
            $CH_2:CHCOOCH_2CX_2CH_3$

NFPMA   2,3-Bis(difluoramino)propyl methacrylate
            $CH_2:C(CH_3)COOCH_2CHXCH_2X$

NFPOH   2,3-Bis(difluoramino)-1-propanol
            $HOCH_2CHXCH_2X$

NP   Nitronium perchlorate
            NO_2ClO_4

OPE   1,2,2,5,9,9,10 - Octakis(difluoramino)-4,7-dioxadecane
            $(CH_2XCX_2CH_2OCHX\cdot)_2$

PETN   Pentaerythritol tetranitrate
            $C(CH_2ONO_2)_4$

PPNF-2   Prepolymer: NFPA/HPMA = 90/10; 0.25% BPO

PPNF-3   Prepolymer: NFPA/HPMA = 90/10; 0.145% BPO

RDX   1,3,5-trinitro-1,3,5-triazacyclohexane
            $\overline{CH_2N(NO_2)CH_2N(NO_2)CH_2NNO_2}$

RETA   Polymer
            [Union Carbide]

TAMA   1,2,4,5-Tetrakis(difluoramino)amyl methacrylate
            $CH_2:C(CH_3)COOCHXCHXCH_2CHXCH_2X$ [Esso]

TEGDN   Triethylene glycol dinitrate
            $(O_2NOCH_2CH_2OCH_2\cdot)_2$

TMETN   Trimethylolethane trinitrate
            $(O_2NOCH_2)_3CCH_3$

TNFH   1,2,5,6-Tetrakis(difluoramino)hexane
            $CH_2XCHXCH_2CH_2CHXCH_2X$

TNFPA   2,2,3-Tris(difluoramino)propyl acrylate
            $CH_2:CHCOOCH_2CX_2CH_2X$

TP-1   TVOPA/PPNF-2 = 2/1

TP-3   TVOPA/PPNF-3 = 2/1

TP-8   TVOPA/PPNF-2 = 3/1

TVOEA   1,1,2,2-Tetrakis(1,2-bis[difluoramino]ethoxy)ethane
$(CH_2XCHXO\cdot)_4C_2H_2$

TVOPA   1,2,3-Tris(1,2-bis[difluoramino]ethoxy)propane
$(CH_2XCHXO\cdot)_3C_3H_5$

RH-P- series propellants are plastisol-nitrocellulose composite formulations.

RH-SA-00 series propellants are non-NF-plasticizer/NFPA-monomer composite formulations.

RH-SA-100 series propellants are TVOPA/NFPA-monomer composite formulations.

RH-SA-200 series propellants are TVOPA/TNFPA-monomer composite formulations.

RH-SB- series propellants are TVOPA/NFPA-HPMA-prepolymer composite formulations.

RH-SC- series propellants are NF-plasticizer/poly(methyl methacrylate) composite formulations.

RH-SM- series propellants are TVOPA/NFPMA-monomer composite formulations.